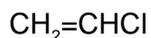


VINYL CHLORIDE

1007



MW: 62.50

CAS: 75-01-4

RTECS: KU9625000

METHOD: 1007, Issue 2

EVALUATION: FULL

Issue 1: 15 February 1984

Issue 2: 15 August 1994

OSHA : 1 ppm; C 5 ppm
NIOSH: lowest feasible; carcinogen
ACGIH: 5 ppm; carcinogen
 (1 ppm = 2.56 mg/m³ @ NTP)

PROPERTIES: BP -14 °C; vapor density 2.2 (air = 1);
 lower explosive limit = 4% v/v in air

SYNONYMS: chloroethylene; chloroethene.

SAMPLING		MEASUREMENT	
SAMPLER:	SOLID SORBENT TUBE (2 tandem tubes, each with 150 mg activated coconut charcoal)	TECHNIQUE:	GAS CHROMATOGRAPHY, FID
FLOW RATE:	0.05 L/min	ANALYTE:	vinyl chloride
VOL-MIN:	0.7 L	DESORPTION:	1 mL carbon disulfide; 30 min
-MAX:	5 L	INJECTION ALIQUOT:	5 µL
SHIPMENT:	separate primary and backup tubes and cap each	COLUMN:	stainless steel, 6.1 m x 3.2 mm, 10% SE-30 on 80/100 mesh Chromosorb W (AW-DMCS)
SAMPLE STABILITY:	10 days @ 25 °C	CARRIER GAS:	He, 40 mL/min
BLANKS:	2 to 10 field blanks per set	TEMPERATURE-INJECTOR:	230 °C
		-DETECTOR:	230 °C
		-COLUMN:	60 °C
ACCURACY		CALIBRATION:	solutions of vinyl chloride in CS ₂
RANGE STUDIED:	1 to 64 mg/m ³ [1]	RANGE:	2 to 200 µg per sample [1]
BIAS:	- 6%	ESTIMATED LOD:	0.04 µg per sample [1]
OVERALL PRECISION ($\hat{S}_{r,T}$):	0.06 [1]	PRECISION (\hat{S}_p):	not determined
ACCURACY:	± 17.8%		

APPLICABILITY: The working range is 0.4 to 40 mg/m³ (0.16 to 16 ppm) for a 5-L air sample. The method is applicable to 15-min samples at concentrations of 1 ppm or higher.

INTERFERENCES: Other than the possibility of loss of sample upon storage of two weeks or more at room temperature, none have been noted.

OTHER METHODS: This is a revision of P&CAM 178 [2].

REAGENTS:

1. Carbon disulfide,* chromatographic quality.
2. Vinyl chloride,*, 99.9%, in lecture bottle fitted with valve and septum.
3. Calibration stock solutions 0.26 mg/mL.
 - a. Insert the tip of a gas syringe containing 1 mL vinyl chloride gas under the surface of 5 mL CS₂ in a 10-mL volumetric flask.
 - b. Open the valve of the syringe and withdraw the plunger to pull CS₂ into the barrel. (As vinyl chloride dissolves, a vacuum will be created, pulling CS₂ into the syringe.)
 - c. Push the solution from the syringe into the flask. Rinse the syringe twice with 1-mL portions of CS₂ and add the washings to the flask.
 - d. Dilute to the mark with CS₂.
4. Helium, purified.
5. Hydrogen, purified.
6. Air, filtered.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: two tandem glass tubes, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends, each containing 150 mg of 20/40 mesh activated (600 °C) coconut shell charcoal. A silylated glass wool plug precedes the charcoal beds and a 3-mm urethane foam plug follows the charcoal beds. Plastic caps are included for sealing after use. Pressure drop across each tube at 1 L/min airflow must be less than 3.4 kPa.
NOTE: A pair of two-section (100 mg/50 mg) tubes may be used. (SKC ST226-01, or equivalent).
2. Personal sampling pump, 0.05 L/min, with flexible connecting tubing.
3. Gas chromatograph, flame ionization detector, integrator and column (page 1007-1).
4. File.
5. Bent wire for removing plugs from sampling tube.
6. Vials, 2-mL, glass with PTFE-lined septa and crimp-on seals.
7. Volumetric flasks, 10-mL, with polyethylene stoppers.
8. Pipettes, delivery, 1.0-mL, graduated in 0.1-mL increments, 2- and 5-mL, with pipet bulb.
9. Air sampling bags, Tedlar, 10-L.
10. Gas syringe, with gas-tight valve, 0.1- and 1-mL.
11. Syringe, 10-μL, with 0.1-μL graduations.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and an acute fire and explosion hazard (flash point = -30 °C); work with it only in a hood.

Vinyl chloride is a human carcinogen [3].

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Break the ends of the tubes immediately before sampling. Attach two tubes, with ends touching, with a short piece of tubing. Label one tube as the back tube and insert the back tube into the flexible tubing attached to the personal sampling pump.
3. Sample at 0.05 L/min for 15 to 100 min. Do not sample more than 5 L of air.
4. Separate the primary and backup tubes and cap each tube for shipment.

SAMPLE PREPARATION:

5. Add 1.0 mL CS₂ to an empty vial. Loosely cap the vial.
6. Score each sampler tube with a file in front of the glass wool plug. Break the tube at the score line.
7. Transfer the charcoal from the front and back tubes to separate vials. Discard the glass wool and foam plugs. Seal the vials with septum caps immediately.
8. Allow to stand for 30 min, with occasional agitation. Analyze the sample within the next 30-min period.

CALIBRATION AND QUALITY CONTROL:

9. Calibrate with at least six working standards covering the range 0.2 to 200 µg per sample.
 - a. Add known amounts of calibration stock solution to CS₂ in 10-mL volumetric flasks and dilute to the marks, using serial dilution as appropriate.
NOTE: Working standards can be stored at -20 °C for at least three days.
 - b. Analyze together with samples and blanks (steps 12 and 13).
 - c. Prepare calibration graphs of peak area vs. quantity (µg) of vinyl chloride per tube and peak area vs. quantity (ng) per injection.
10. Determine desorption efficiency (DE) at least once for each lot of charcoal used in the calibration range (step 9). Prepare three tubes at each of five levels plus three media blanks.
 - a. Prepare three atmospheres of vinyl chloride in air by injecting 0.01, 0.08, and 0.2 mL vinyl chloride gas into 10 L air in Tedlar bags. The resulting concentrations are approximately 2.6, 21 and 52 mg/m³.
 - b. Following steps 1 through 4, sample these atmospheres according to the following scheme:

Concentration in Bag (mg/m ³)	Volume Sampled (L)	Quantity of Vinyl Chloride (µg)
2.6	0.8	2
	2.2	6
21	0.8	17
	2.2	46
52	2.5	130

- Obtain three samples at each level.
- c. Desorb (steps 6 through 8) and analyze together with working standards (steps 12 and 13). No vinyl chloride should be found on the back tubes.
 - d. Analyze the atmospheres in the bags (steps 12 and 13) using 1-mL gas samples to verify concentration.
 - e. Prepare a graph of DE vs. µg of vinyl chloride recovered.
11. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are in control.

MEASUREMENT:

12. Set the gas chromatograph according to manufacturer's instructions and to conditions given on page 1007-1. Inject sample aliquot manually using solvent flush technique or with autosampler. The retention time of vinyl chloride is about 1.7 min.
NOTE: If peak area is above the linear range of the working standards, dilute with CS₂, reanalyze and apply the appropriate dilution factor in calculations.
13. Measure peak area.

CALCULATIONS:

14. Determine the mass, μg (corrected for DE) of vinyl chloride found in the sample front (W_f) and back (W_b) tubes, and in the average media blank (B).
NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
15. Calculate concentration, C, of vinyl chloride in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - 2B)}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

The method was evaluated with single 150-mg coconut shell charcoal tubes (100-mg front beds and 50-mg back) [1]. Atmospheres were generated at four concentrations between 1 and 64 mg/m^3 . Recoveries, based on atmosphere concentrations calculated from the volumes of vinyl chloride and dilution air, averaged 94% with a pooled relative standard deviation (\hat{S}_{rT}) of 0.06. Samples at the 3- μg level showed no loss of vinyl chloride when stored for 12 days at room temperature or 19 days at -20 °C. There may be significant loss of vinyl chloride from samples stored for 14 days at room temperature [4]. The 1% breakthrough capacity for a 150-mg bed of coconut charcoal, challenged at 100 mL/min with vinyl chloride in air at 16 mg/m^3 and a relative humidity of 70%, was 4.6 L [5].

REFERENCES:

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